EXHIBIT 81

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MINERALOGICAL TECHNIQUES OF ASBESTOS DETERMINATION

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BARBEAU – CHEMICAL METHODS

Dupuis, M. (1974): Caractérisation atomique et moléculaire du chry-
    sotile des mines d'amiante de l'Estrie. Thèse de maîtrise,
    Université Laval.

Harington, J.S. (1965): Chemical studies of asbestos. Ann. N.Y.
    Acad. Sci. 131, 31-47.

Harris, A.M. and Grimshaw, R.W. (1975): The leaching of ground chry-
    sotile. Abstracts, third international conference on the
    physics and chemistry of asbestos minerals, Québec, 1975,
    paper 4.17.

Holt, P.F. and Clark, S.G. (1960): Dissolution of chrysotile
    asbestos in water, acid and alkali. Nature, 185, 237.

Monkman, L.J. (1971): Some chemical and mineralogical aspects of
    the acid decomposition of chrysotile. Abstracts, second inter-
    national conference on the physics and chemistry of asbestos
    minerals, Louvain, 1971, paper 3.2.

Morgan, A., Holmes A. and Lally, A.E. (1971): Solubility of chryso-
    tile asbestos and associated trace metals in N hydrochloric
    acid at 25ºC. Abstracts, second international conference on
    the physics and chemistry of asbestos minerals, Louvain, 1971,
    paper 2.8.

Papirer, E., Dovergne, G. et Leroy, P. (1976): Modifications physi-
    co-chimiques du chrysotile par attaque chimique ménagée: I- En
    milieu aqueux. Bull. Soc. chim. (5-6), 651-3.

Pundsack, F.L. and Reimschussel, G.P. (1956): The properties of
    asbestos: III Basicity of chrysotile suspensions. J. Phys.
    Chem. 60, 1218-22.

SECTION 6

METHODS FOR THE EVALUATION OF ASBESTOS DUST CONCENTRATION AT THE

WORKPLACE

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INTRODUCTION

This section, although directed more toward industrial hygiene
than toward mineralogy, is included in order to arouse interest
amongst mineralogists to the problem of evaluating chrysotile asbes-
tos dust concentration. The need for further development in the
field of asbestos fiber determination is also emphasized.

The objectives pursued, the strategies elaborated by industry
and the instrumentation used for the examination of air-borne asbes-
tos dust at the workplace will first be discussed. Some of the
methods presented here do not measure the fibers but rather, the
respirable dust; we will see how these methods nevertheless conform
to industrial strategies, within certain limitations. Many methods
and many instruments will perhaps be omitted in this text. We will
explain only the instrumentation made available to the Quebec Asbes-
tos Mining Association.

It should be noted that every subject covered in this text may
be found more completely explained in the literature. Therefore,
the bibliographical reference list, while not exhaustive, should be
of assistance to the interested reader.

DEFINITIONS

Some words, which are frequently encountered in the text, may
not be clearly understood or may lack specification because of their
universal usage. For this reason, I have chosen to re-explain a few
key words in the sense in which they are understood by the industrial
hygiene community. The minerals themselves, such as chrysotile,
antigorite, lizardite, serpentine, will not be mentioned since they
should be known to the reader.

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is of particular interest to the plant engineers. Already we can
see the need for instruments that are automatic, autonomous, fast,
accurate, robust and of low maintenance requirements.

The spot-checking strategy permits the evaluation of the effi-
ciency of new designs in the maintenance program and also permits a
quick evaluation of the operation as a whole in a mine and in a mill.
This strategy supports continuous monitoring and the compliance pro-
grams by readily detecting priority areas, 'machinery wise'. It per-
mits quick detection of the sources of dust emission, their impor-
tance and their influence on other sections of activity in the plants.
The instrumentation needed here must be automatic, light (readily
portable), fast, easy to operate and autonomous for several hours.

Fixed station sampling for asbestos fiber dust is useful to the
manager who needs information to quantify his compliancy with the
standards set up by regulating agencies. This kind of sampling does
not give information on the exposure of the worker but it does measure
the industry's effort to reduce the exposure of workers to toxic
dust. Thus, this strategy gives the lowest levels of dust to which
the workers are exposed - or the background concentration of asbestos
dust in a mill.

The exposure of workers to asbestos dust is measured by the next
strategy - personal sampling. This involves the evaluation of two
additive parameters: Job habits and the performance of the industry.
The engineering control people need this information in order to pro-
duce proper job habits among their labor force. The medical people
can operate only with this method, in order to make the correct dose-
response relationship towards the toxicity of the dust.

There are other strategies and other objectives that we did not
talk about. Training of personnel, specific control of one type of
equipment, research and health in the neighborhood of the workplace
are all objectives which require the elaboration of strategies and
that create a need for other specific types of instrumentation.

THE CHOICE OF AN INSTRUMENT

New instruments come on the market every week. An instrument
which is the best available today for one strategy may become obso-
lete tomorrow. The industry must remain alert to technical develop-
ments. Moreover, all new types of instrumentation should be inves-
tigated by the asbestos industry, even if there is no immediate need.
The technical people should always know what is available in order
to better devise feasible strategies to attain their objectives.
The development people must also realize that a laboratory instrument
is not always immediately suited to the industry, but may often
require design improvements.

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Although we know that the following day may bring a new instru-
ment to better fulfill a strategy, we must take action when action
is needed and not wait indefinitely.

Let us remember that the main objective is not to comply with
a standard or to measure dust levels, but rather it is to reduce the
dust level as much as possible in order to insure health at the work-
place.

INSTRUMENTATION

Numerical concentration (f/cm^3)

The membrane filter method. This method of sampling, mounting
and counting fibers is used both for strategies of fixed station
sampling and for personal sampling. It does have some limitations.
However, they have been created mainly by misinterpretation on the
part of some people, and by a free attitude of varying important
parameters by others.

Many regulating agencies or bureaus (Bayer and Zumwalde,1973;
AIHA-ACGIH,1975; Australian Dept. of Health,1976; ARC,1971; AIA,1978)
have proposed variants of this method. All, nevertheless, agree on
one essential description:

"The sample is collected by drawing a measured quantity of air
through a membrane filter by means of a battery powered sampling
pump. The filter is later transformed from an opaque membrane to a
transparent, optically homogeneous specimen. The fibers are then
sized and counted using a phase contrast microscope and the concen-
tration of fibers in the air is expressed as fibers per millilitre
of air, calculated from the number of fibers on the filter and the
measured volume of air sampled". (AIA,1978).

The apparatus used to measure dust by the so-called 'membrane
filter method' permits only an index value of the real concentration
of asbestos fibers in air. The index concentration is based on the
measurement of particles with a 3:1 (length/diamter) aspect ratio,
which are longer than 5μm, whose diameter is limited by the respi-
rability of the fiber at one end (3.0μm) and by the resolution of
the microscope used at the other end (≈0.5μm). This index is accu-
rate enough for workplace application, since it is sensitive to
different conditions.

Gibbs (1978) has stated that, based on transmission electron
microscope observations, the percentage of fibers that would be ob-
served by optical microscopy is between 1.9% and 10.6% for the
chrysotile textile industry. These percentages may vary with the
type of industry but they show that it is important to know exactly
what counting method was used before any number is interpreted.

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Problems with the method arise when analysing its precision and the cause of errors or differences in various fiber counts. Under one generic name, many membrane filter methods exist; it is not an exaggeration to say that there are as many methods as there are independant laboratories. Even within a laboratory, different microscopes may create an unwanted, significant difference between two counts. (Deadman, J. and Pellerin, N., 1978)

These differences, which could be avoided by the standardization of the membrane filter method, come mainly from slight variations introduced by different laboratories. The parameters of the method, listed below (table 6.1), are suspected of being among the main sources of difference.

Table 6.1: Variations in dust counts due to differences in techniques within the membrane filter method.

- Sampling	- the flowrate
	- the velocity at the input of filter
	- the diameter of the filters
- Mounting	- the mounting liquids
- Counting	- the counting criteria
	- the objectives
	- the phase contrast system
	- the graticules (if any)
	- the magnifications

There are also some errors which are inherent to the method and others, which are due to human interference (Table 6.2).

Table 6.2: Causes of variation in dust counts due to errors inherent in the method.

- Sampling	- variation in the flowrate of one pump
	- non-uniform distribution on the filter
	- recording of sampling time
	- contamination
- Mounting	- the quantity of mounting liquid may create the condition of migration of the particles
- Counting	- the different comprehension of the same criteria of counting by two technicians.
	- the 'state of mind' of one technician

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Because of these causes of difference and error within the membrane filter method, it is believed that the coefficient of variation of the counting step within one group of laboratories is ±15% when all technicians are using the same microscope fields (Trudeau,1978d). Finally, the method can become very imprecise (100 to 1) when inexperimented technicians operate it. (Walton,1977)

In order to conserve the credibility of the method, the Asbestos International Association (AIA), through its Dust Measurement Advisory Panel (DMAP), has recently proposed a uniform membrane filter method to the industry (AIA,1978). This method, inspired from the Australian membrane filter method, has adopted the best from all existing methods, in order to attain the highest possible precision. Some parameters are still under study; others could not be changed or overly restricted due to the specific instrumentation which is already available, but an effort was made toward the understanding of all parameters of sampling, mounting and counting fibers as indices to health conditions at the workplace.

The method is presently being examined by an international team of experts that look critically at the proposal; a final proposition will be then made that contains as few as feasible multiple choice parameters (filters,microscopes,...). Using this method, comparative data between laboratories may become available. Entitled, "Draft proposal of a reference method for the determination of fiber concentration at workplaces in the asbestos industry by optical microscopy" (AIA,1978), it seems judicious to present its essential schemes here below. (Figure 6.4)

This presentation is not the accepted AIA universal method, but it is sufficient for the reader to understand its main implications. Anyone wishing to use the method itself should go to the Asbestos International Association and obtain a complete copy of the proposal.

Sampling: It is not within the scope of this text to explain in great detail the various strategies of sampling since this text is directed more towards analysis than to sampling itself. Briefly, the strategies of sampling may vary from 'fixed station sampling', where the sampling pump is mounted on a tripod for a period depending on the actual estimated concentration of dust and on the flowrate of air through the filter, to 'full shift personal sampling', where the flowrate is adjusted in relation to the expected dust cloud. The various 'in-between' strategies depend on the possibilities within the industry, on the apparatus used and on the specific objectives pursued.

Filter: Membrane filters (mixed esters of cellulose or cellulose nitrate) of 0.8μm pore size with printed grids and diameters of 13 to 37mm should be used. (Millipore AAWG03700 at QAMA)
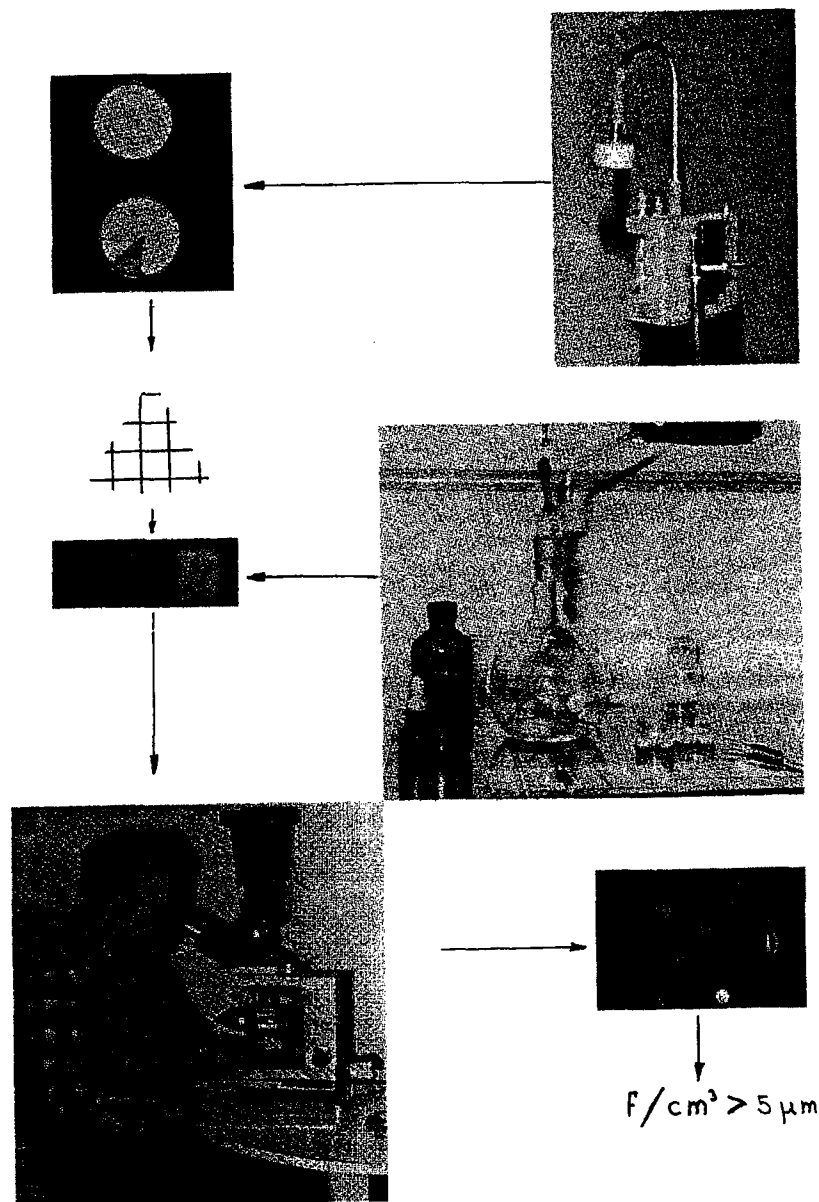
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Fig. 6.4.   A schematic presentation of the membrane filter method.

$$f/cm^3 > 5 \mu m$$

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Filter holder: For 25mm and 37mm diameter filters, use open faced filter holders fitted with a protective cowl. These protect the filter from accidental contamination.

Transport: The filter may be transported with its filter holder. Fixing fibers to the filter surface is not necessary. If it is inconvenient to transport a full filter, the samples may be shipped already mounted or taped to the bottom of a Petri dish.

Sampling pump: The capacity of the battery must be sufficient to operate continuously over the chosen sampling time. The flow must be free from pulsation such that visible vibration of the membrane filter is not present (Damping criteria is yet to be established). An external damper may have to be installed between the pump and the filter.

Flowrate: The flowrate has to be adjusted so that a face velocity in the ranges between 1 and 5cm/sec can be obtained. (This criteria is being further investigated).

The flowrate has to be determined with an installed or external flowmeter and has to be checked at least before and after sampling. In case of long-term sampling the flow must also be checked during the sampling period. The average flowrate is used for evaluation. The sampling has to be repeated if the initial and final flowrates differ by more than ±10% from each other.

The flowmeter used must be calibrated by using a soapfilm flowmeter, wet test meter, spinometer, or similar device at the selected flowrate. The sampling train used in the calibration (pump, tubing, filter and filter holder, etc...) should be the same as that used in the field. The calibration equipment and technique should be of such accuracy that the flowrate can be measured to within±5%.

Sampling period: The sampling period should be adjusted so that the concentration per surface area (mm^2) on the filter permits a precise evaluation. The sampling time may be adjusted for a maximum of 3 to 5f/graticule area depending on the type of dust sample and the area of the graticule used.

Mounting the sample: All the apparatus used should be thoroughly clean. A dirty working area may result in sample contamination and erroneous results.

The 13mm diameter filter can be mounted completely. When using 25mm and especially 37mm diameter filters, it is recommended that a piece with an area of about 1 square cm be cut off with a scalpel. Usually the cut piece has a 'pie' shape.

There are two methods of mounting a sample using the so-called acetone+triacetin method. Only the one used by the QAMA will be presented here. One is reminded to use great care when working with

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acetone. On no account should acetone be used in the vicinity of an open flame.

Heat the acetone and wait until a moderate quantity of acetone vapour is emerging from the outlet of the boiling flask (250 or 500ml). Ensuring that no liquid acetone drops on the filter (by wiping the outlet with a tissue periodically), hold the filter with clean forceps directly in the acetone vapour, approximately 15-25mm from the outlet, for 1-3 seconds. Move the filter slowly across the outlet to ensure even coverage. Too little vapour will fail to render the filter transparent, while too much vapour (especially drops of liquid acetone) will destroy the filter by dissolving it or shrinking it beyond use. The slide must not be prewarmed, as the acetone vapour must condense on the slide for correct clearing.

After leaving the acetone cleared filter to stabilize at least 10 minutes, lay a clean coverslip on a horizontal surface and using a hypodermic syringe with a 22 gauge needle, place a small amount of glycerol triacetate (Triacetin) on the coverslip. Lower the already cleared filter on to the coverslip and press gently with forceps to ensure even spread of the Triacetin. Too much Triacetin (as indicated by excess liquid filling the space between coverslip and slide for more than approximately 2mm outside of the filter area) can cause the outside edge of the filter to eventually disintegrate to some degree. One or two drops is often sufficient. Insufficient Triacetin will result in uneven clearing of the granularity left from the acetone vapour clearing. Furthermore, the refractive index of the mounted sample will not be suitable for optimum visibility of very fine chrysotile fibers.

Triacetin, when it becomes very old starts to decompose and forms acetic acid. Since this acid attacks chrysotile fibers, Triacetin with any noticeable "vinegar-like" odor should not be used. It is sometimes necessary to delay counting for up to 24 hours until the entire filter has dissolved under the action of the Triacetin. However, the finished product will be stable, will not disintegrate, nor be subject to particle migration. Nail polish, or similar lacquer, must be painted around the edge of the coverslip if the slide is to be kept indefinitely.

Counting the slide: It is recommended that the following specification be used to select a microscope suitable for asbestos dust counting. This specification is similar to that of the Asbestosis Research Council (1971b). Koehler illumination is essential. It is preferable for the illuminator to be built-in but an external lamp with a plane mirror can be satisfactory. A variable intensity control is necessary for both methods of illumination. The Koehler illumination assures a uniform illumination of the object by mixing the rays of light. It is necessary that the source of light be reproduced in the back focal plane of the objective and that each point of the source illuminates all of the object plane. (Determann and Lepusch, 1969)

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Substage Assembly: An Abbe or achromatic phase-contrast condenser incorporated into a substage unit is recommended. There must be a means of centering the diaphragms with respect to the corresponding phase plate in the objectives and a means of focusing the condenser.

Stage: A built-in mechanical objective stage fitted with verniers and slide clamps is required.

Objectives: A rotating nose piece with 10x and 40x parfocal phase-contrast achromatic objectives is essential. For purposes of standardization between laboratories, the 40x objective must have a numerical aperture (NA) of 0.65. It should have a phase ring of 90% absorption. Either positive or negative phase contrast is suitable.

Eyepieces: Binocular eyepieces of the compensating type are recommended. They should be chosen to give a total magnification of between 400x and 640x. At least one eyepiece should permit the insertion of a graticule.

Graticule: The graticule counting area must be between $0.0025mm^2$ and $0.016mm^2$. Figure 6.5 shows the several satisfactory graticules. (Further work is being conducted to investigate whether the above specifications should be tightened).
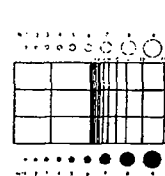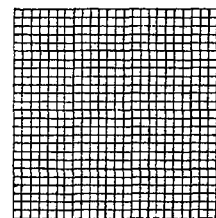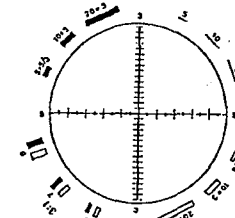

a) Porton
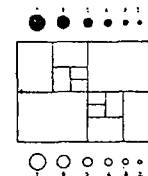
b) BS 3625

c) German
($125\mu m^2$, $5 \times 5\mu m^2$ mesh)

d) Walton & Beckett
($100\mu m$ diam)

Fig. 6.5. Several graticules used for fibers counting with an optical microscope.

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A centering telescope or Bertrand lens is essential for checking that the phase rings in the condenser are centered with respect to those in the objective. Green or blue filter is essential to ensure the best phase contrast conditions. Stage micrometer for calibration must be sub-divided into 10μm intervals and preferably should be 1mm long. The best quality microscope slides are (0.8 to 1.0mm thick) glass slides.

Coverslips are a necessary part of the slide mount and optical system. A convenient size is 22mm x 50mm; these can be handled by the corners without contaminating the field view. Most microscope objectives are optically corrected for a no. 1½ (0.17mm thick) cover-slip. Different cover-glass thickness will detract from the quality of the final image.

Microscope adjustment: The image of the light source must be in focus and centered on the condenser iris or annular diaphragm for true Koehler illumination. The object for examination must be in focus. The illuminator field iris must be in focus, centered on the sample, and opened only to the point where the field of view is illuminated. The phase rings (annular diaphragm and phase shifting elements) must be concentric. The eyepiece graticule must be in focus. Preferably the eyepiece should have adjustable focusing. It is suggested that microscope adjustments become a daily routine.

Eyepiece graticule calibration: Each combination of eyepiece, objective and graticule must be calibrated with a stage micrometer. Should any of the three be changed, the combination must be recalibrated. For some microscopes, calibration will change for observers with different interpupillary distances.

Counting and sizing fibers: Air-borne asbestos dust collected on membrane filters appears in a wide variety of forms ranging from simple single fibers to very complex configurations of fibers and aggregates. When presented with these, the microscopist could experience difficulty in defining and counting the fiber content of a dust sample.

When counting and sizing, constant use of the fine focus control is necessary because of the small depth of focus of a 40x objective (i.e. 2-3 micrometers). Counting fields should be chosen systematically across the filter. Ensure that the choice of fields is not biased by lack or presence of fibers. Do not select fields that lie within 3mm of the filter edge and within 2mm of the cutting line.

Designate all particles having the following geometric dimensions as fibers:
  – length greater than 5 micrometers, and
  – diameter less than 3 micrometers, and
  – length to diameter ratio greater than 3:1

Be as accurate as possible in accepting or rejecting fibers close to 5 micrometers in length and 3 micrometers in diameter. Estimate the length of curved fibers along the curve. When examined microscopically, asbestos fibers fall into four basic groups as follows:
  – Single fibers can be directly classified according to their geometric dimensions.
  – Split fibers are counted as 1 fiber.
  – Grouped fibers of width <3μm are counted as 1 fiber. If >3μm, they are not counted.
  – Fibers attached to particulate matter are counted as one fiber if the particles is <3μm, otherwise they are not counted.

Count all fibers within the graticule area. If a fiber crosses any side of the graticule area count half a fiber if the end is inside the graticule area. Do not count any fiber which crosses two sides (Cooper et al., 1978).

If more than one sixth of the counting field contains an agglomerate of fibers and/or dust, reject the field and select another. Always record such occurences.

Count as many fields as is necessary to yield a total fiber count of 100 but count a minimum of 40 fields even if more than 100 fibers are counted. Do not count any more than 100 fields if a total of 100 fibers is not reached. Errors will arise if these numbers of fibers and fields are not counted.

Calculation of dust concentrations: The fiber concentration is determined according to the formula and is valid only for the period over which the sample was taken.

$$C = \frac{A}{a} \cdot \frac{N}{n} \cdot \frac{1}{r} \cdot \frac{1}{t}$$

where:
C – concentration (fibers/cm^3)
N – total number of fibers counted
n – number of graticule areas counted
A – effective filter area (mm^2)
a – graticule area (mm^2)
r – flowrate of air through filter (cm^3/min)
t – sample duration (minutes)

If several consecutive samples are used for characterizing the workplace, a time weighted average value should be calculated from single values:

$$ C = \frac{\sum C_i . t_i}{t_i} = \frac{C_1 t_1 + C_2 t_2 + \ldots\ldots + C_k \quad t_k}{t_1 + t_2 + t_3 \ldots\ldots\ldots + t_k} $$

where:

C – time weighted average value

C_i – single value of concentration

t_i – time for which C_i is representative

k – total number of samples

THE FAM (Fibrous Aerosol Monitor)

The FAM, made by GCA Corporation/Technology Division, is the only instrument now available on the market that monitors the concentration of fibers numerically; as such, it may, after calibration, replace the membrane filter method in some of its applications.

The best brief description of the FAM may be found in the advertisement brochure of GCA Corporation/Technology Division (1977). A few additions and corrections have been made to the original text, since the FAM presently on the market has had several recent design improvements.

"The air driven by a diaphram pump enters the FAM at a rate of 2 L/min and passes through a chamber in which laminar air flow is developed. The air is then drawn through the sensing region where the particles are exposed to the rotating electric field (3000V/cm peak). This field induces a dipole charge separation on the fibers and aligns them with the applied rotating field. This, in turn, imparts to the fiber a motion of several hundred oscillations per second.

Concurrent with its movement through the sensing chamber, each particle is illuminated by a continuous wave helium–neon beam (632.2nm) aligned parallel to the direction of air flow. The resulting light scattering pulses are detected by a photomultiplier tube located along the side of the sensing chamber. This detector measures the light scattered perpendicular to the axis of the laser beam. The angular dependence of the light scattered by cylindrical particles can be compared with a rotating searchlight beam, or with the emission of a pulsar, in that the detected signal is very sharply peaked at the maxima and nearly zero at all other angles. In fact, it is the degree of this signal pulse sharpness that serves as the basis for the method of fiber length discrimination used on the FAM, since longer fibers produce narrower pulses than shorter ones.

The electronic circuitry of the FAM applies four different acceptance tests to every detected signal and only if all of these

criteria are met is that signal train registered as a discrete fiber count: (a) the signal pulses must be synchronous in frequency and phase with the internal reference waveform, (b) the average amplitude of the pulses of a train must exceed a preset minimum, (c) the average ratio of the central to the side portions of the pulses must exceed a preset minimum and (d) the duration of the pulse train must exceed a preset minimum.

The technique of induced fiber oscillations and concurrent detection of the resulting light scattering signature results in a powerful discriminatory method for the selective counting of airborne fibers even in the concomitant presence of other, non fibrous, particles in concentration exceeding those of fibers by factors of up to 10^6.

Following the completion of the sample cycle, the FAM automatically computes the concentration of fibers in units of fibers per cubic centimeter and displays this value on a liquid crystal display.

The basic FAM unit runs off a.c. line power (115 or 220 volts), (50 or 60 Hz) but incorporates a modular design concept permitting the use of a battery powerpack available as an accessory. In addition, either analog or digital recorders are available as optional accessories permitting long-term continuous monitoring." (GCA/Technology Division, 1977b)

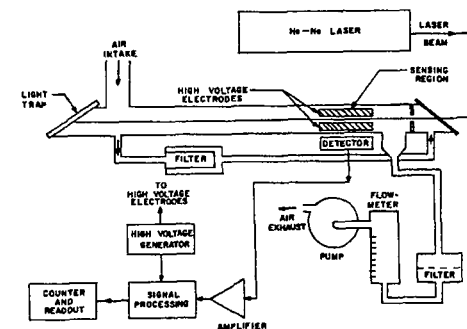Figure 6.6 shows schematically the operation of the machine.

Fig. 6.6. Schematic of optical and flow systems of FAM. (by permission of GCA Corporation/Technology Division).

Counting periods from 1 minute to 1000 minutes, increasing by geometric increment of 10, permit the measurement of concentrations ranging from $0.0001f/cm^3$ to $20f/cm^3$. The estimated minimum detectable fiber diameter is $0.2\mu m$ (Lilienfeld,1978). Weighing about

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13.6 kilograms (30 pds), its dimensions are 53cmx33cmx16cm.

The FAM was recently the object of an investigation by the Quebec Asbestos Mining Association (Trudeau,1978e). The experiment was designed to test the capacity of the apparatus in measuring significantly fibrous dust in the chrysotile asbestos mining indus- try and to evaluate the need for the FAM within the strategy frame- work explained above.

Two sets of membrane filters were installed parallel to the FAM and later measured. One membrane filter was located near the air entry of the FAM, the other was located at the output of the FAM where a plenum was installed in order to distribute the dust uni- formly on the filter. The two sets of membrane filters proved to be equivalent (coefficient of correlation $\approx$ 0.86). From then on, only the first set (air entry) was used. A series of 30 comparative samples were taken throughout the mines and mills, not only in various locations but also in various dust clouds.

The FAM was not calibrated. The samples were taken doubly using two sets of calibrations, hoping that one set would give values in the same order as the membrane filter values. At ratio 4 (calibra- tion knob on the FAM), the data were quite comparable. Table 6.3 shows a few results.

Table 6.3:   Linear regression of FAM data (ratio 4) in relation to the Membrane filter results. $(Y = A_1 X + A_0)$

Y	X	Companies	N	A_1	A_0	r
FAM4	MF	11	7	.19	.09	.87
FAM4	MF	21	9	1.06	1.13	.93
FAM4	MF	32	9	.61	1.67	.84
FAM4	MF	all	30	.92	.66	.82

N = nb of samples
r = correlation coefficient

The disparity of the slopes (A_1) between the three companies shows that the calibration of the FAM is sensitive to different dusts. We must admit though that the apparatus was subjected to very difficult situations and that the analysis did not take into account the reproducibility of dust counting by the Membrane filter method. Nevertheless, the apparatus has proven, without a doubt, that it is able to give the information for which it was designed. The exact manner to perform the calibration and how frequently this must be done, should be investigated more thoroughly. It was shown that the apparatus is sufficiently sensitive to any typical "mineral" dust and that it is not at all influenced by gases or smokes issued from underground blasting.

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The FAM may be used as a research tool to define qualitatively the granulometric configuration of fibrous dust within one mine or within one location. By using the "ratio" knob of the FAM, which controls sensitivity to the lengths of the fibers (Lilienfeld,1978), one may obtain qualitative information on the length of the fibers at several locations. It was found that at low concentrations, the length distribution of the fibers is quite constant within one mine or one mill, since the data using two ratios were highly correlable. Table 6.4 shows a few results.

Table 6.4:   Relation between FAM results at different positions of the "ratio" knob. $(Y = A_1 X + A_0)$

X	Y	Companies	N	A_1	A_0	r	X	Y
FAM5	FAM4	11	7	1.68	.01	.87	1	1.69
FAM5	FAM4	21	9	1.69	.83	.92	1	2.52
FAM5	FAM4	32	7	2.36	.51	.86	1	2.87

N = nb of samples
r = correlation coefficient
X = FAM values at position 5 on ratio knob
Y = FAM values at position 4 on ratio knob

Table 6.4 also shows that when the measurements were done, the dust in the air at mine 32 was finer than the dust at the two other mines. At high, non-typical concentrations, the fibrous dust does not follow any model; this is to be expected when the dust cloud is due to secondary dust source resulting from accidental situations.

Finally, an evaluation was made to determine the need for the FAM within the industry. As the strategies of the QAMA are now out- lined, the FAM could find an application only in fixed-station sam- pling or spot-checking the dust for engineering control. Spot-checking is presently taken care of by other automatic, autono- mous gravimetrical instruments.

For fixed-station sampling, the FAM would save membrane filter counting time. It is worthy to note that the FAM does not pretend to obtain better precision than the membrane filter method. Routine use of the FAM would be more time-consuming than use of the membrane filter method. For example, if one desires a significant evaluation of the dust cloud within one location, the 100 minute sampling time should be used. The other periods available are 10 and 1000 minutes. A technician may handle one FAM at a time. The usual fixed station sampling period by the membrane filter method is 90 minutes.

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A technician is able to handle 3 or 4 sampling pumps at a time.
The mounting and counting time may be approximated at 30 minutes.
Therefore, 3 samples by the FAM would take 300 minutes, whereby
3 samples by the membrane filter method would take 180 minutes.

Other applications will assuredly be found for the FAM.
Also, different agencies may incorporate this apparatus into their
own strategies. In addition, the FAM may be used for monitoring the
dust, an aspect which was not considered here, because of other ins-
tallations (see APM). However, within the industry, the future of
numerical evaluation probably resides with automatic counting of
membrane filters (Taylor, C.J., 1978).

Gravimetrical concentration (mg/m³)

Several instruments will be presented that are used rou-
tinely by the QAMA member industries. There are many other instru-
ments, which work on similar principles and which are now available
on the market, that are not mentioned here. Also, other instrumen-
tation that could be useful in dust measuring employing different
strategies than those previously enumerated will not be mentioned.
This text should not be considered as a blind endorsement of one
manufacturer's products against its competitors.

APM (Ambient Pollution Monitor)

Fig. 6.7. The APM

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Made by GCA Corporation/Technology Division, the APM was
designed to measure automatically the dust concentration in the
general environment using the β–radiation attenuation principle.
Its sensitivity, autonomy and sturdiness permitted us to employ the
APM for monitoring of returned air in the asbestos mills. Lilienfeld
(1975) discusses in detail the β–radiation attenuation principle for
measuring the dust. For low energy radiations, the absorption is
mainly a function of the electronic density of the absorbant.

The electronic density may be expressed as:

$$\rho_e = \rho \cdot N \cdot \frac{Z}{M}$$

where:
ρ_e – Electronic density
ρ – Mass density
N   – Avogadro's number
Z   – Atomic number
M   – Mass number

Except for hydrogen (Z/M=1), the ratio of atomic number to mass
number varies for all the elements from 0.4 to 0.5. By setting the
calibration of an instrument for an average Z/M=0.45, the maximum
deviation will be minimized and the electronic density becomes a
linear function of the mass density, regardless of the chemical
composition.

Errors resulting from this method are due primarily to fluctua-
tion of the source, variations in the density of the air between the
source and the detector during the measurement period and presence
of inherent radioactivity in the absorbants. Avol and Clark (1976),
in a previous test, have mentioned that the APM response is linear
in relation to the concentration of dust and that it is independant
of the composition of the dust.

The apparatus is composed of three modules:

The sampling-sensor module collects the dust and evaluates its
concentration. The APM samples on two channels simultaneously; one
channel may be equipped with a dust separator and the other without.
In our mills, we generally measure respirable dust as separated by
a 10mm cyclone on one channel and total dust on the other channel.
(figure 6.8)

This module simulates a high volume collector; an axial flow
pump assures a flowrate of 1690 L/min (60cfm) through a duct within
which the sample is taken at a rate of 9 L/min. The flowrate of the
sampling system is kept constant with a venturi type passive control.
Also, in order to prevent condensation, an electric heater assures
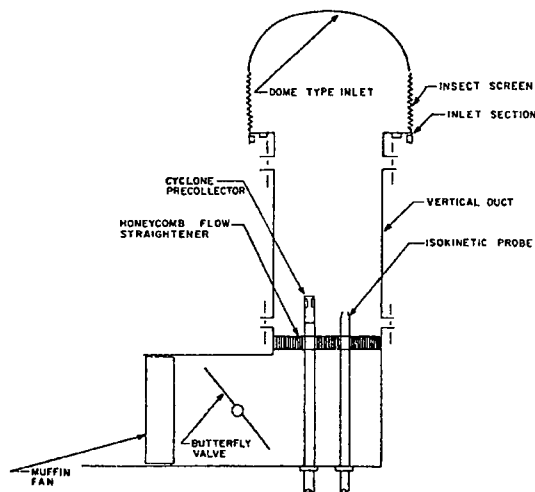a variation of $\Delta T = 30^{\circ}F$ in the temperature of the sampled air.

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Fig. 6.8.   APM inlet manifold design sketch. (with permission
of GCA Corporation) (GCA Corporation, 1977)

The dust is collected on a fiberglass filter reinforced with
cellulose. The radiation sources are ^{14}C and the sensors are Geiger-
Müller counters. ^{14}C has a half-life of 5730 years and an energy of
radiation of 0.156MeV, which fulfills the double condition of low-
energy and penetration capacity. The design of the machine is such
that, the same spot on the filter is used twice; one spot is measured
before and after the sampling time with the same pair of source-
sensor. In order to do this, the filter moves in alternation, one
step backward and two steps forward.

Figure 6.9 illustrates the #1 filtration spot: At step 1 the
spot is pre-analysed for the first time, for background measurements.
At step 3, a sample is taken. At step 4, the post-analysis is made
and the result appears at the end of step 5. Then, the pre-analysis
is made on the same spot by the second Geiger-Müller counter. At
step 6, the sample is taken and at step 8 the post-analysis is made.
At the end of this same step the result appears for the second ana-
lysis on this same spot. The great advantage of this design is that
the filter lasts twice as long. The print-out of the measurement
is always ready after a period of time equal to twice the sampling
time. The sampling time may be adjusted to between 2 minutes and
999 minutes, according to the expected concentration. For example,
an expected concentration of 100µg/m^3 (0.1mg/m^3) would be sampled

for 30 minutes for an evaluation at ±20% (with 95% degree of con-
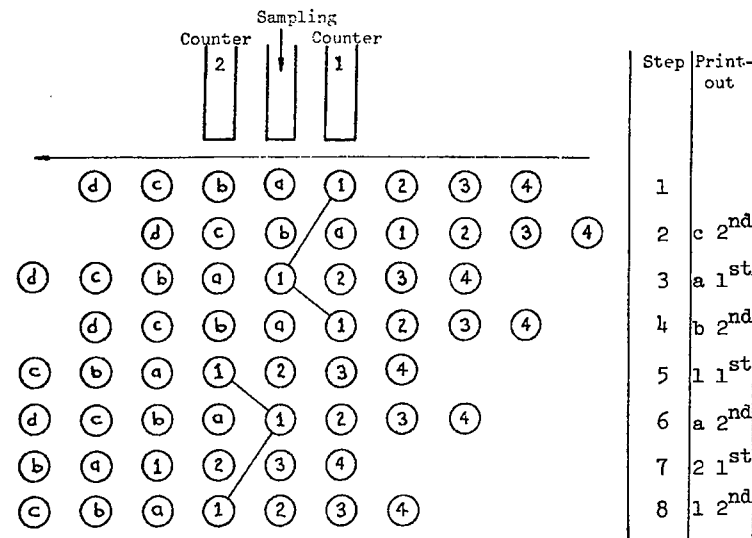fidence).

Fig. 6.9.   Movement of a filtration spot on the APM.

The electronic module does the treatment of the data. The
results are automatically registered on a digital paper print-out.
Each time, the date, the time and the duration of sampling are
written on the two channels called R and T, in addition to the
results expressed in µg/m^3.

The computation of the mass concentration is performed by the
calculation of the following equation:

$$C = \frac{A}{\mu Q t} \quad (\ln F_o - \ln F) \quad \mu g/m^3$$

where:
- C – concentration
- A – surface of sampling
- Q – flowrate (m^3/sec)
- t – duration of sampling
- F_o – counts before sampling
- F – counts after sampling
- μ – absorption coefficient (cm^2/µg)

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As for the calibration, one must only check the blank counts regularly to make sure that the pairs source-detectors are performing correctly. The need for further calibration is obviated because of the design of the sampling-sensor module. Finally, the electronic system is equipped with multiple sensors that give information on any malfunction of the apparatus.

The pump is the mechanical module. It is designed to pump 9 L/min of air whatever the mass of dust on the filter. Naturally, when a certain level is attained, clogging occurs and the electronic module shows a flow fault (FLF). The pump is very stable, even when the voltage fluctuates.

APM vs Standard gravimetric evaluation: Several measurements with the APM were paralled with a gravimetric method using the PVC Metricel filters. Figure 6.10 shows the 21 comparative results on respirable dust taken in 4 different mills. The dust separators used were the 10mm cyclones. Each APM value is an average of many measurements. A good correlation exists ($\rho=0.91$) between the two instruments and the regression line is very close to the line of slope 1.
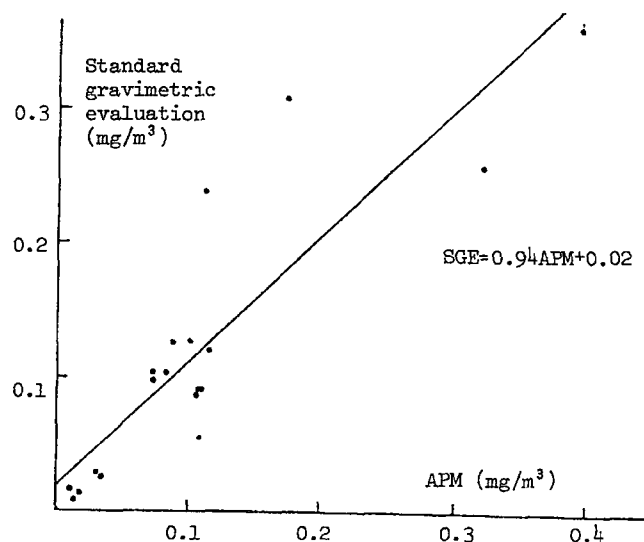
Standard gravimetric evaluation (mg/m^3)

SGE=0.94APM+0.02

APM (mg/m^3)

Fig. 6.10. Respirable dust analysis. APM:Standard gravimetric evaluation.

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APM data - an example: The sampling time choosen by the companies varies from 35 minutes to 2 hours depending not only on the effective concentration of the dust but also on the cycling time of the bag filters system. The long sampling time (2 hours) gives more accuracy on a single measurement ($2\sigma \approx 2.1\mu g/m^3$), but the results cannot be easily used by the maintenance people since the concentration value is printed only 4 hours after the end of the sampling period. A short sampling time (35 minutes) is individually imprecise ($2\sigma=9\mu g/m^3$), but the information on unusual behavior of the control system is more readily available (70 minutes) and the precision on a 24-hour average is very close to the precision on a 24-hour average using 2 hours sampling time $2\sigma_{35} : 2\sigma_{120}$ (1.4.:0.7). The only drawback of the short sampling time is the occasional awkward negative results when the real concentrations are low.
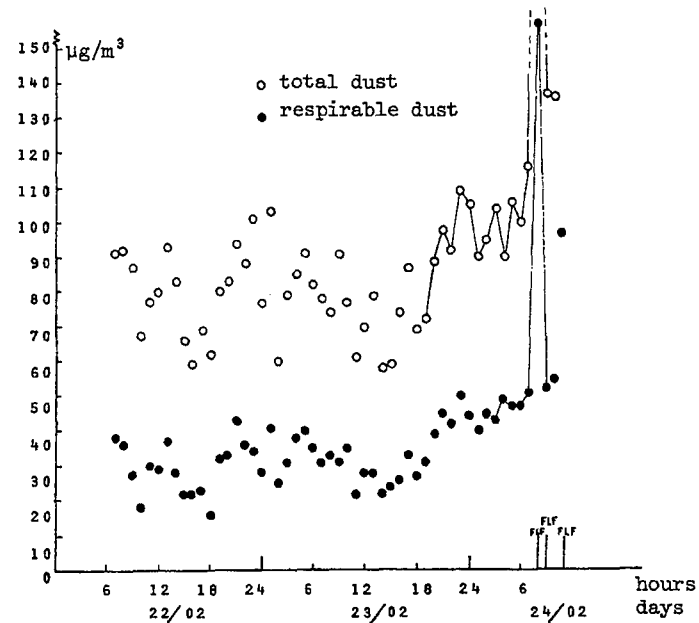
o total dust
• respirable dust

Fig. 6.11. Dust Measurement:APM data.

Because the design is set up so that a spot is used twice, the system may show a clogging occurrence (flow fault) the second time the spot is sampled. This may only signify that a difficult condition occurred when the spot was first sampled. On the other hand, the sampling time may be adjusted so that no flow fault will occur. The flow fault signal indicates either a high concentration causing a clogging of the filter or a leak between the venturi and

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the filter. When the sampling time recorded is higher than 10 minutes, we usually take for granted that the data is significant; when the sampling time is inferior to 10 minutes, the data is not included in the computations, but shown as a flow fault occurrence.

Figure 6.11 shows results typical of those drawn from an APM tape, or similar to what we could get on an analog output. The data were taken in the general plenum of a typical mill. When a mill is in operation the respirable dust values are about 30% of the total dust values. We can see that the concentration of respirable dust varies between 16 and 50µg/m^3 (.016 to .050 mg/m^3). The respirable dust curve and the non-respirable dust curve are parallel, showing either that the dust control efficiency is not a function of the granulometry of the dust or that the dust itself has a constant granulometric distribution. It is noteworthy to mention that the concentration showed a trend before the incident which occurred at the eight hour of the 24/02. (An incident is defined as a non-normal value, a trend or by any break in the series of values).

RDM 101-4.

The Respirable Dust Monitor RDM 101-4, made by GCA Corporation/ Technology Division, is a light-weight automatic instrument used mainly for "spot-checking" the dust in the asbestos mills. (figure 6.12) It is used by hygienists in order to get a quantitative measure of the concentration of the dust in terms of mg/m^3 in an area of a mill or a mine. The RDM 101-4 is also used with success in environments of silica, cotton and other types of dust (Lilienfeld, 1972; Almich et al.,1975; Neefus,1974). The RDM 101-4 collects respirable dust at a rate of 2L/min by impaction on a Mylar disc on which a layer of grease (4 parts vaseline, 2 parts parafin oil); has been spread.
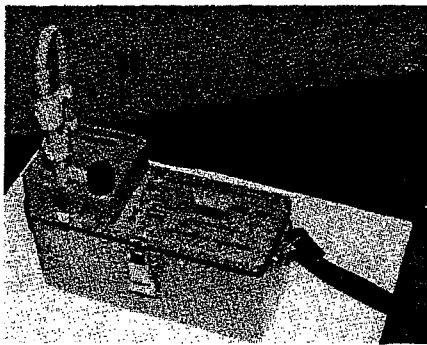

Fig. 6.12. The RDM 101-4

At the QAMA, the non-respirable fraction of the dust is separated by a 10mm nylon cyclone. This cyclone passes all the particles with an aerodynamic diameter less than 2µm   (Lilienfeld and Dulchinos, 1972). A preliminary study (Archambault, 1978) about the behavior of the separators in an asbestos environment showed that for a low dust mass (≈1.1.mg) the nylon cyclone followed very well the ACGIH model curve for respirable dust.

The measurement of the concentration is performed automatically with the β-radiation attenuation principle. (see paragraph on APM). Figure 6.13, borrowed from Marple (1976), shows the head of the apparatus. The β-radiation emitter is ^{14}C; a Geiger counter is used as a sensor. The effective diameter of the dust spot is approximately 0.66mm.
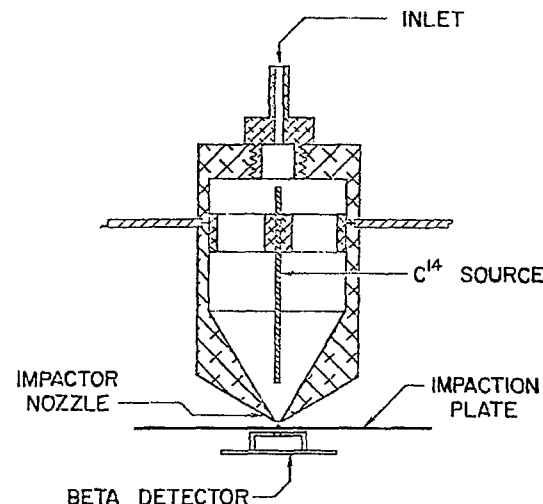

Fig. 6.13 RDM 101-4 inlet assembly (Marple, 1978)
(with permission of AIHA Journal).

The RDM 101-4 can be run on the automatic mode for concentrations of 0.02 to 50.0mg/m^3. The sampling time is 220 sec. and the initial and final counting are done during the first and final 20 seconds of the measuring cycle, which is 240 seconds (4 minutes) long. For concentrations lower than 0.02mg/m^3, it is better to use the instrument on the manual mode and for a longer sampling period.

The dust concentration can be read directly on the digital output of the apparatus. A polyester disc may be used for 90 samples before replacement becomes necessary. The RDM 101-4 runs autonomously for about 4 hours.

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It is noteworthy to mention that the impactor does not stop particles with an aerodynamic diameter inferior to 0.4μm; for rock dust ($d \approx 2.4$ g/cm^3) it would be 0.21μm (Lilienfeld and Dulchinos, 1972). This may become an advantage when one wants to avoid too sensitive a response to cigarette smoke or to another very fine aerosol.

RDM 101-4 vs standard gravimetry. The GCA company claims a precision of $2\sigma = \pm 25\%$ on each measurement when compared to the true concentration as measured with a standard gravimetric instrument. We could not verify this statement in the QAMA mills because of the large dust variations over the period of time necessary to sample the dust in the standard manner. Nevertheless, the averages obtained with the RDM 101-4 correlate very well with the values obtained with standard gravimetric instruments (Metricel PVC filter + 10mm cyclone). The similarity of results depends on the calibration of the RDM 101-4. Several tests were performed in different mines.

Table 6.5 shows the results using two distinct apparatus in two different mines.

Table 6.5: Comparative measurements: the RDM 101-4 vs the Metricel + 10mm Cyclone. ($Y = A_1 X + A_0$)

Company	Slope of Regression line (A_1)	A_0	Number of Data	Correlation Coefficient
51	1	0	15	0.99
21	1.36	.03	12	0.97

y = RDM 101-4 values (average over 14 measurements)
x = Metricel + 10mm cyclone

RDM 101-4 and the membrane filter method. No direct relationship was found between the RDM 101-4 data and the membrane filter method data from the mines (Trudeau, 1978c). However, we can attempt to use the RDM 101-4 data to suggest compliance to the values of 2f/cm^3>5μm by using the technique illustrated in figure 6.14. Teichert (1978) and Knight (1977) first introduced this concept to the asbestos mines. In this technique, a vertical line is drawn at 5f/cm^3 and a horizontal line drawn such as to set equal numbers of pairs of measurements in the upper left quadrant (7 pairs) and lower right quadrant (7 pairs). This gives a value of 0.85mg/m^3 equivalent to 5f/cc>5μm. With these two criteria, the two sampling systems agree in placing 108 samples as being

either below or above the respective standards and disagree on 14 samples, or 11%.
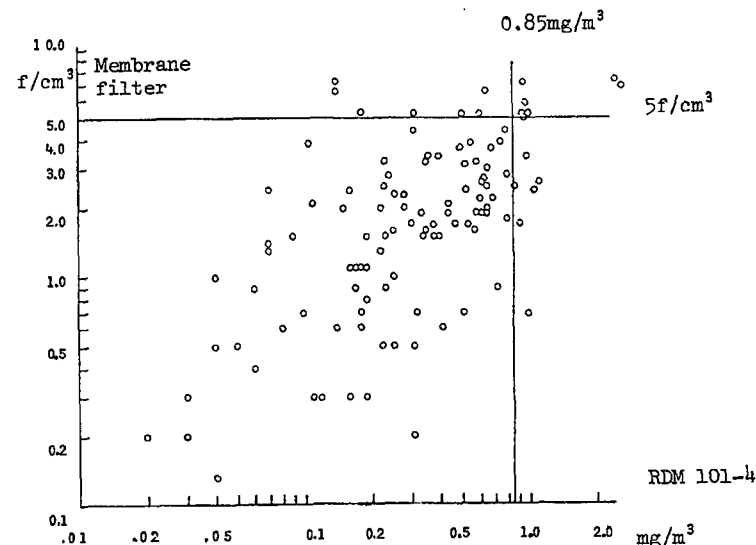

Fig. 6.14. Determination of equivalent dust concentrations.

Typical results are given on Table 6.6. Note that these values were taken throughout several mills on one occasion. It may happen that they will change in another survey or that the relationship is more reliable when the different steps in the processing of the ore are treated separately.

Table 6.6: Respirable mass and the number of fibers. Equivalence.

Company	Concentration (mg/m^3) equivalent to		Agreement % at	
	2f/cc	5f/cc	2f/cc	5f/cc
11	.16	0.23	82	85
12	.20	0.30	85	87
21	.50	2.0	90	100
51	.43	1.0	89	98
4	.35	0.87	85	93

(from Knight, G. (1977): Gravimetric sampling in Asbestos Mines and Mills, Nov., QAMA Technical Report).

THE TYNDALLOMETER TM DIGITAL

The Tyndallometer, manufactured by Ernst Leitz Wetzlar, is designed for in-site measurements. It may be used as a monitoring or as a spot-checking instrument. (figure 6.15)
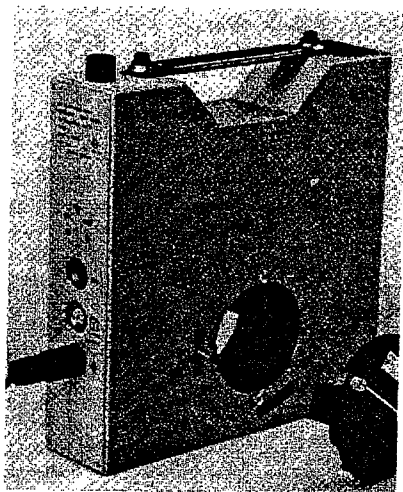

Fig. 6.15. The Tyndallometer

The Tyndallometer, as its name suggests, puts into application the "Tyndall" effect: "whenever microscopic particles, that have dimensions comparable to the wavelength of light, are present in a transparent environment, their presence is not detected by transmission, as they do not produce a shadow, but by laterally diffracted light; this phenomenon is called diffusion of light". (translated from Fleury et Matthieu, 1968, p.66)

The intensity of diffusion of light is primarily a function of four parameters:
- the dimension of the particles
- the total volume of particles (number of particles)
- the wavelength of the incident beam
- the angle between the direction of the incident beam and the scattered light.

For a constant volume of dust and for a monochromatic incident beam, the intensity of the diffusion increases with the fineness of the particles and decreases with the angle of diffusion.

The Tyndallometer was designed so that the signal measured is proportional to the volume of dust deposited in the lungs; the signal is then converted to gravimetrical terms (mg/m^3), using the average density of the measured particles. The Tyndallometer is insensitive to particles larger than $10\mu m$ diameter.

With asbestos fibers, the intensity of diffusion is dependant on the direction of the fibers; however it is assumed that the measurements will be representative of an average orientation, since the dust is measured in place without any disturbance.

The measurement of the diffusion is made at an angle of 70° with the incident monochromatic beam ($\lambda=0.94\mu m$). These conditions were set up to optimize sensitivity to respirable dust and to permit a large range of concentrations ($0.01mg/m^3$ to $99.9mg/m^3$). Tests made in the U.S. (Tomb, Treaftis and Thompson, 19) showed a correlation coefficient of 0.98 in mines, when the Tyndallometer results were compared to the measurements made with approved dust samplers. Breuer and Robock (1975) demonstrated that the influence of temperature between 0^oC and 40^oC is minimal; the apparatus is always calibrated at 20^oC.

Dust accumulated on the lens of the Tyndallometer does not affect the results as much as dust accumulated on the sides of the dust chamber (figure 6.16). This is due to the undesired reflection of light through the silicon photodiode detector. It is essential that the chamber be thoroughly clean. This may be easily verified by checking the zero calibration of the apparatus. The Tyndallometer enables short-time measurements, within 10 sec., or even real time measurements by the use of continuous recording.

Usage of the Tyndallometer. It is possible to use the Tyndallometer telemetrically with analog output to monitor the dust in baghouses, in-stacks, etc... In the QAMA, the Tyndallometer is used as a spot-checking instrument, to give valuable information on the variations of dust concentration.

A dust cloud may vary its composition or its granulometric distribution very rapidly, especially in the case where incidents occur. Cigarette smoke and humidity affect the readings. Therefore, use of the absolute values from a Tyndallometer is very limited. However, the Tyndallometer does have a great advantage: its capacity to give very fast results. It is an essential tool for maintenance people looking for bag filters leakages or other incongruous dust sources. The Tyndallometer has also become a valuable tool for testing new bag filters, since it was found to be more sensitive to slight variation in dust concentration than is the membrane filter method.

In other types of asbestos industries (Teichert, 1978), the Tyndallometer is used to examine dust variations in relation to the

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operations performed. This allows the production of information helpful to the engineer who is setting-up his priorities in dust control.
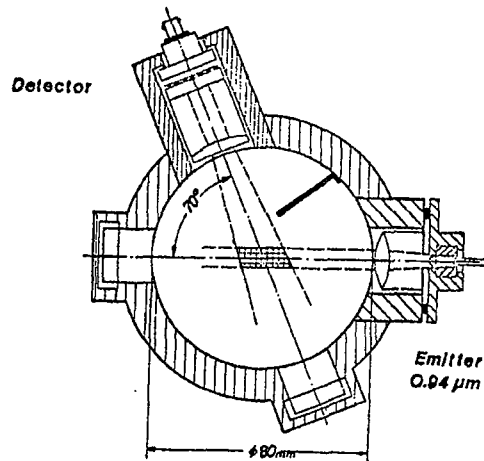

Fig. 6.16. The Tyndallometer measuring chamber (from AIA,1977)

CONCLUSION

This presentation has outlined the instrumentation presently being used by the QAMA, in order to perform dust concentration evaluation at the workplace. The analysis of asbestos dust is a very difficult task. From the definition of a fiber to the final interpretation of the measurements, most of the subjects treated could generate discussions and, even, polemics.

All the methods presented have limitations: some lack accuracy, others precision, some are tedious or need specialists.

Many instruments, which were omitted, would probably be suited for use within the same framework. The development of different strategies and of better techniques to measure dust will eventually render today's modern instrumentation obsolete. It is hoped that the technology available in the future will permit the elaboration of strategies that comply more fully to their objectives.

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REFERENCES

AIHA-ACGIH (1975): Recommended procedures for sampling and counting asbestos fibres. Procedures for the evaluation of occupational exposures to airborne asbestos. Amer. Ind. Hyg. Assoc. Journ., Feb., pp 83-90.

Almich, B., Solomon M. and Carson, G.A. (1975): A theoretical and laboratory evaluation of a portable direct-reading particulate mass concentration instrument. HEW publication. NIOSH, 76-114.

American Conference for Governmental Industrial Hygienists (1968): Threshold limit values of air-borne contaminants for 1968, Cincinnati.

American Conference of Governmental Industrial Hyegienists (1977): TLVs Threshold limit values for chemical substances in work-room air adopted by ACGIH for 1977.

Archambault, George (1978): Etude des séparateurs de poussière dans les moulins d'amiante. Avril, Rapport interne AMAQ, 99p.

Asbestos International Association (1977): International colloquium on dust measuring technique and strategy, Warmensteinach, Germany. August 29-30. (Report can be obtained from Wirtschaftsverband Asbestzement E.V., Görlitzer Strasse 1, D-4040 Neuss, Deutschland).

Asbestos International Association – Dust Measurement Advisory Panel (1978): Draft proposal of a reference method for the deter-mination of fibre concentration at workplaces in the asbestos industry by optical microscopy. Second Colloquium on Dust Measuring Technique and Strategy. Oct. 11-13, Washington.

Asbestosis Research Council (1971a): Technical note 1: "The measu-rement of airborne asbestos dust by the membrane filter method". September. Rochdale. Lancashire.

Asbestosis Research Council (1971b): Technical note 2: "Dust sam-pling procedures for use with the asbestos regulations 1969. January. Rochdale. Lancashire.

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